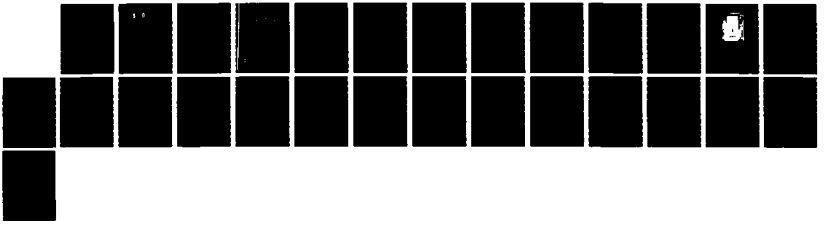
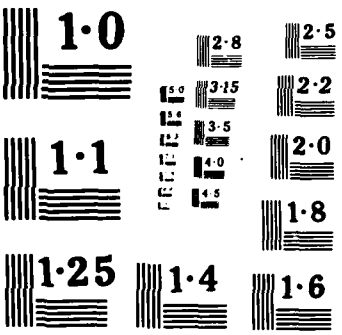
AD-A170 001 STUDY OF HIGH TEMPERATURE FAILURE MECHANISMS IN CERAMICS(U) SOUTHWEST RESEARCH INST SAN ANTONIO TEX DEPT OF MATERIALS SCI.. R A PAGE ET AL. APR 86 1/1

UNCLASSIFIED SWRI-8578/2 AFOSR-TR-86-0485 F/G 11/2 NL




1·0
1·1
1·25
1·4
1·6
1·8
2·0
2·2
2·5
2·8
3·15
3·5
4·0
4·5

AD-A170 001

REPORT DOCUMENTATION PAGE

1b. RESTRICTIVE MARKINGS	
2a. SECURITY CLASSIFICATION AUTHORITY	3. DISTRIBUTION/AVAILABILITY OF REPORT
2b. DECLASSIFICATION/DOWNGRADING SCHEDULE	Approved for release; distribution unlimited
4. PERFORMING ORGANIZATION REPORT NUMBER(S)	5. MONITORING ORGANIZATION REPORT NUMBER(S)
SwRI-8578/2	AFOSR-TR- 86-0485
6a. NAME OF PERFORMING ORGANIZATION: Southwest Research Institute, Dept. of Materials Sciences	6b. OFFICE SYMBOL (If applicable)
7a. NAME OF MONITORING ORGANIZATION: Air Force Office of Scientific Research, Electronic & Materials Sciences Directorate	
6c. ADDRESS (City, State and ZIP Code): 6220 Culebra Rd. (P.O. Drawer 28510), San Antonio, TX 78284	7b. ADDRESS (City, State and ZIP Code): Bolling AFB, Building 410, Washington, DC 20332
8a. NAME OF FUNDING/SPONSORING ORGANIZATION	8b. OFFICE SYMBOL (If applicable): NE
9. PROCUREMENT INSTRUMENT IDENTIFICATION NUMBER: F49620-85-C-0073	
8c. ADDRESS (City, State and ZIP Code): Bolling AFB DC 20332-6448	10. SOURCE OF FUNDING NOS.

PROGRAM ELEMENT NO.	PROJECT NO.	TASK NO.	WORK UNIT NO.
61102F	2306	A2	

11. TITLE (Include Security Classification): Study of High Temperature Failure Mechanisms in Ceramics (unclassified)

12. PERSONAL AUTHOR(S): Page, Richard A., Lankford, James

13a. TYPE OF REPORT	13b. TIME COVERED	14. DATE OF REPORT (Yr., Mo., Day)	15. PAGE COUNT
Annual	FROM 4/1/85 TO 3/31/86	1986, April	25

16. SUPPLEMENTARY NOTATION

17 COSATI CODES			18. SUBJECT TERMS (Continue on reverse if necessary and identify by block number)
FIELD	GROUP	SUB. GR.	

19. ABSTRACT (Continue on reverse if necessary and identify by block number)

This report summarizes the results of a fundamental study involving experimental characterization and analytical modeling of grain boundary cavitation and creep crack growth in structural ceramics exposed to pure tensile loading. The major experimental techniques employed in the program are the use of small-angle neutron scattering to characterize cavity nucleation and growth and stereoimaging analysis to characterize the stress and strain fields associated with growing creep cracks.

In the first section of the report, the experimental progress is summarized. The design of the pure tensile creep apparatus, which is being used for the creation of bulk damage and for creep crack growth, is discussed. The progress made in the determination of surface preparation conditions that are adequate for the stereoimaging analysis is also discussed.

(continued)

20. DISTRIBUTION/AVAILABILITY OF ABSTRACT	21. ABSTRACT SECURITY CLASSIFICATION
UNCLASSIFIED/UNLIMITED X SAME AS RPT ☐ DTIC USERS ☐	Unclassified

22a. NAME OF RESPONSIBLE INDIVIDUAL	22b. TELEPHONE NUMBER (Include Area Code)	22c. OFFICE SYMBOL
Major Joe Hager	202/767-4984	AFOSR

The second section of the report describes the results of a critical review of recent experimental and theoretical studies of creep cavitation in ceramics. The results of this critical study have identified a number of stochastic aspects of cavitation. The stochastic nature of cavitation arises primarily due to the dependence of both cavity nucleation and cavity growth on grain boundary sliding. A degree of randomness is also imposed by the nonuniform distribution of nucleation sites. These results suggest that the measurement of grain boundary sliding rates and the development of a statistical model of cavitation will be crucial to the understanding and modeling of tensile creep failure.

STUDY OF HIGH TEMPERATURE FAILURE MECHANISMS IN CERAMICS

By
Richard A. Page
James Lankford

AFOSR ANNUAL REPORT

This research was sponsored by the Air Force Office of Scientific Research,
Electronic and Materials Sciences Directorate
Under Contract F49620-85-C-0073

Approved for release; distribution unlimited.

April 1986

SOUTHWEST RESEARCH INSTITUTE
SAN ANTONIO HOUSTON

SOUTHWEST RESEARCH INSTITUTE
Post Office Drawer 28510, 6220 Culebra Road
San Antonio, Texas 78284

STUDY OF HIGH TEMPERATURE FAILURE MECHANISMS IN CERAMICS

By
Richard A. Page
James Lankford

AFOSR ANNUAL REPORT

This research was sponsored by the Air Force Office of Scientific Research,
Electronic and Materials Sciences Directorate
Under Contract F49620-85-C-0073

Approved for release; distribution unlimited.

April 1986

Approved:

Gerald R. Leverant, Director
Department of Materials Sciences

TABLE OF CONTENTS

LIST OF FIGURES

I. RESEARCH OBJECTIVES

1. Utilize small-angle neutron scattering to characterize cavity nucleation and growth rates under tensile creep conditions as functions of time, temperature, stress, strain, strain rate, and microstructure.
2. Measure experimentally the local strains, grain boundary displacements, and displacement rates attendant with the above mentioned cavity characterizations.
3. Incorporate the measured cavity nucleation and growth rates and the local deformation measurements into a model for grain boundary cavitation under tensile creep.
4. Characterize creep crack growth and experimentally measure, using stereoimaging strain analysis, the crack-tip displacement field, strain singularity, and creep strain rates as functions of stress intensity, temperature, microstructure, and precavitation level.
5. Incorporate the cavitation model and measurements and the crack-tip micromechanical measurements into a fundamental creep crack growth model for ceramics.

II. STATUS OF THE RESEARCH EFFORT

A. Scope

Because of the attractive strength properties of ceramics at elevated temperatures, there is great interest in developing a new generation of aerospace propulsion systems capitalizing on advanced ceramics technology. These new propulsion systems would potentially offer higher operating temperatures and lower weights, thus providing dramatic increases over current engine designs in both efficiency and performance. While present engines utilize hot-stage components fabricated from nickel or cobalt-base superalloys, it is anticipated that evolving ceramic turbines will be based on silicon nitride and silicon carbide. In service, the ceramic components will experience tensile and/or cyclic loadings. Very little is known, however, about the behavior of these ceramics under tensile creep or cyclic creep conditions. An understanding of the basic failure mechanisms and an ability to predict lifetimes will be necessary before ceramics can be successfully utilized in engine applications.

In order to understand creep failure of ceramics, several specific issues must be addressed. These include: (1) characterization of the development of creep cavities at grain boundaries in bulk specimens as a function of tensile stress; (2) characterization of grain boundary displacement during bulk creep; (3) characterization of crack tip stress relaxation due to crack tip creep strain; (4) characterization of cavity distributions ahead of creep cracks; (5) performance of pure tensile tests

at elevated temperatures; (6) creation, and characterization of the growth, of creep microcracks. The progress made along these lines during the initial year of the program is summarized below.

B. Current Status

Usually, "tensile" testing of ceramics has been performed in flexural bending, a compromise which produces a stress gradient across the specimen. Pure tensile testing of brittle materials is difficult, because of alignment considerations which can produce unknown, unwanted bending moments, hence spurious strength measurements. However, it is important to the goals of the proposed program to achieve pure tensile testing, since (1) it is desired to characterize cavitation in terms of a known applied tensile stress normal to the cavitating grain boundaries, and (2) it is necessary to secure a reasonably large sample of material cavitated at a uniform stress level for the SANS measurements. Bend tests violate both of these requirements.

A major portion of the first year of this contrast was thus spent designing and fabricating a tensile creep apparatus. A schematic of the tensile creep frame is presented in Figure 1, and a close-up photograph of the environmental chamber, furnace, specimen, and gripping arrangement is presented in Figure 2. This frame utilizes deadweight loading and is capable of testing at stresses in excess of 1000 MPa and temperatures up to 2300°C. Self-aligning universal joints are affixed to the specimen grips, which, acting in conjunction with the flexures that are machined into both the upper and lower pull rods, minimize the bending moment imposed on the sample during testing.

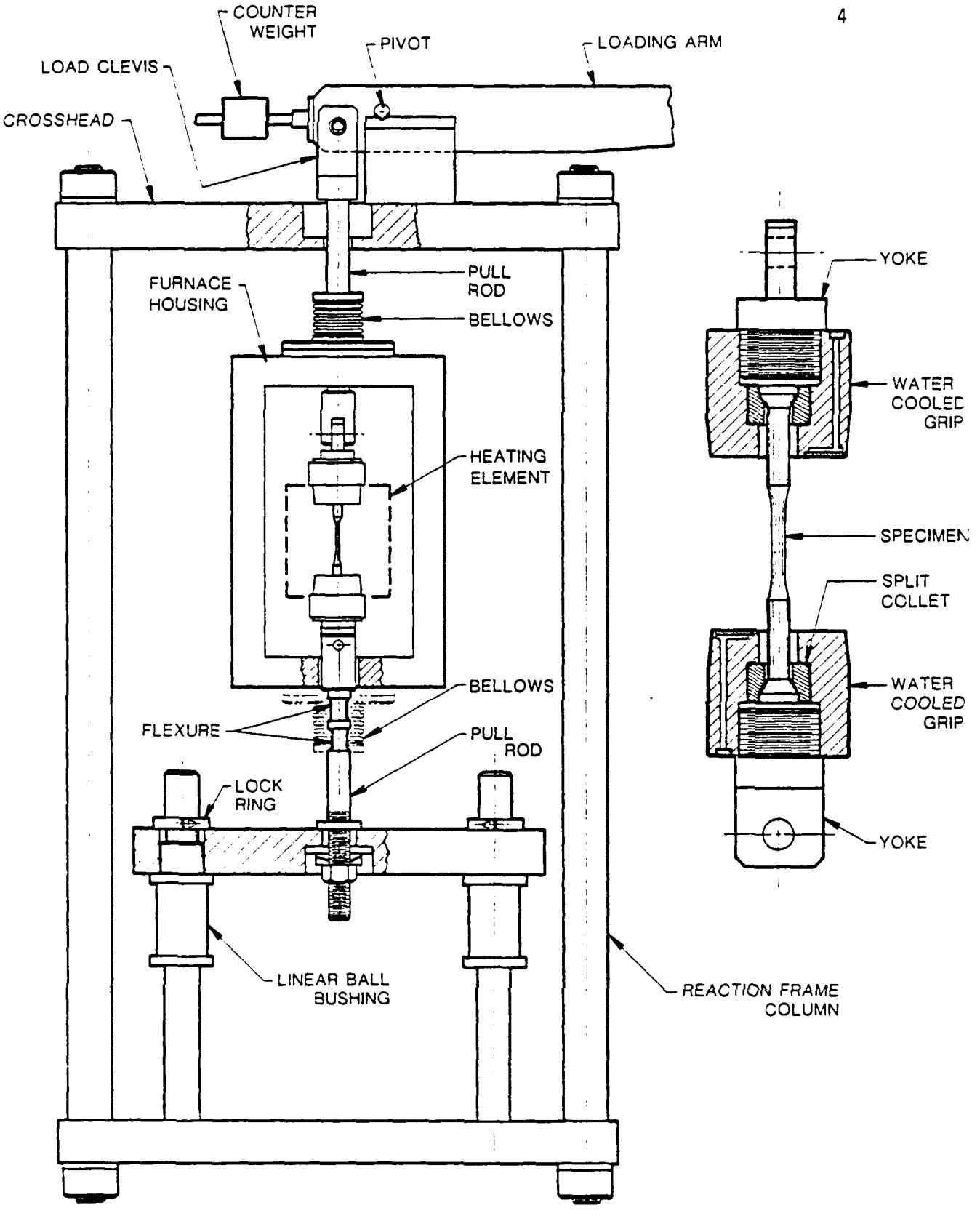


FIGURE 1. SCHEMATIC OF TENSILE CREEP FRAME. Cut-away view showing the gripping arrangement and the specimen geometry on the right.

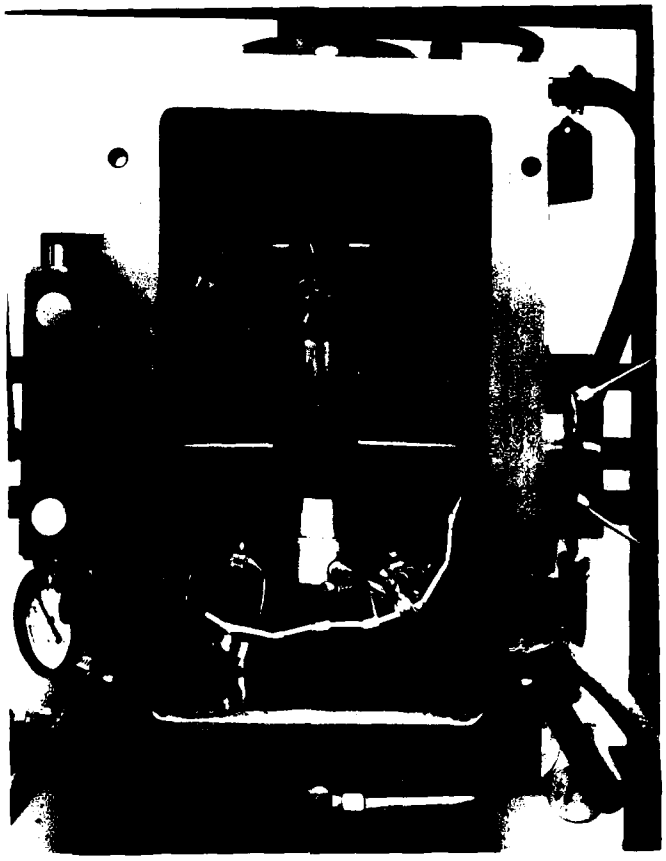


FIGURE 2. PHOTOGRAPH OF THE ENVIRONMENTAL CHAMBER OF THE TENSILE CREEP FRAME SHOWING THE FULLY ASSEMBLED GRIPPING ARRANGEMENT. Also visible are the back halves of the tungsten heating elements and the heat shields.

Close-up views of the gripping assembly and specimen design are also shown in Figure 1. The grip assembly is composed of a water-cooled superalloy main body which encloses split ceramic collets. Since ceramic specimens cannot relax plastically at grip contact points like metal alloys, boron nitride powder is used as a powder cushion lubricant between the specimen ends and the collet-type grips. The specimen design, which is based on finite element stress calculations, minimizes stress concentrations in the gage and grip areas.

Surface preparation techniques for the stereoimaging strain analysis were also examined during the first year. Generally, the stereoimaging analysis requires two photographs of the same region obtained at different deformation states; for creep loading this is accomplished by photographing the same area at various times. In this manner, the strains and strain rates can be determined. Since the analysis is a surface technique, it requires that the surface remains unmodified, apart from modification due to the creep strains, during the time interval between the photographs. Examination of the ceramic to be used in the initial series of tests, Norton NC 203 hot-pressed silicon carbide, showed that polished surfaces were substantially modified during thermal holds at temperatures around 1600°C. Thus, as-polished surfaces are not sufficient for the stereoimaging measurements. Investigation of a number of alternate surface preparation techniques indicated that surfaces that were given a thermal etching treatment following polishing did not change dramatically during subsequent creep or thermal treatments. It thus appears that polished and thermally etched surfaces will be sufficient for the stereoimaging measurements.

In summary, the first year of this contract was spent developing the capability to perform pure tensile creep on structural ceramics and determining the proper surface preparation techniques for the stereoimaging analyses. While both of these took a longer period of time than anticipated, we feel that we are now in a position to rapidly move ahead on the characterization of bulk creep damage and creep crack growth in the SiC and Si_3N_4 materials during the second year of the program.

C. Stochastic Aspects of Cavitation

While the tensile creep frame was being constructed, the substantial body of data in the literature pertaining to creep cavitation was carefully studied with the intent of identifying the critical parameters in cavitation. Although most of the quantitative data were obtained from compression tests, the basic concepts, at least, should be applicable to the tensile experiments of the present study. The results of this critical study, which are briefly summarized below, have formed the basis of a manuscript titled "Stochastic Aspects of Creep Cavitation in Ceramics", which is currently being prepared.

Previous studies of compressive creep in both silicon carbide (1-3) and alumina (3-6) have demonstrated that cavity nucleation definitely takes place during creep of ceramics. As shown in Figure 3, cavity nucleation is frequently continuous. Furthermore, the high cavity density indicates that cavitation occurs on two grain facets since there are insufficient three grain facets to account for the high cavity density observed. The presence of cavities on two grain facets has been confirmed by TEM (3,5) and SEM (6). To explain the nucleation of cavities along two grain facets Chan, et al (7) have postulated that nucleation occurs at

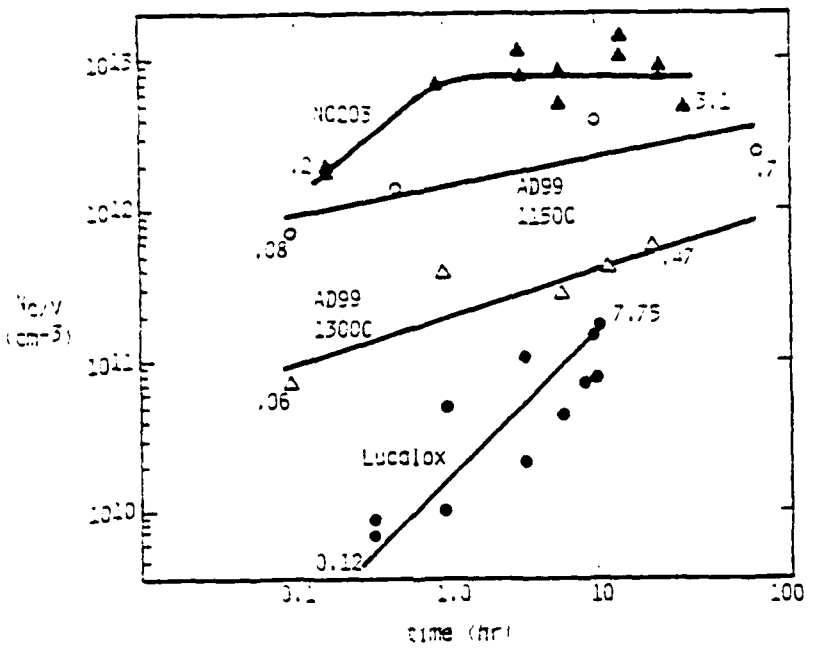


FIGURE 3. NUMBER OF CAVITIES PER UNIT VOLUME VERSUS TIME. Cavity density was determined by small-angle neutron scattering from compression crept ceramics.

ledges present along the boundary. As shown in Figure 4, grain boundary sliding would produce a high tensile stress concentration at the ledges. However, grain boundary diffusion would act to remove the stress concentrations. Thus, if ledges are to act as efficient nucleation sites, nucleation must occur prior to the diffusive relaxation of stress at the ledges.

Chan, et al (7) have calculated the various relaxation times and compared them to estimates of the incubation time for cavity nucleation. The results of these calculations, Figure 5, suggest that a narrow range of h/λ, where h is the ledge height and λ is the ledge spacing, exists in which nucleation is expected. As demonstrated in Figure 6, at large values of h/λ the stress concentration relaxes prior to nucleation, while at small values of h/λ the stress concentration never builds up due to inhibited sliding. It is only in the central regime of h/λ that stresses of sufficient magnitude are present for a long enough duration to result in cavity nucleation. The above discussion identifies two critical parameters pertaining to cavity nucleation, grain boundary sliding and the grain boundary microstructure, both of which are stochastic in nature.

Estimates of cavity growth rates based on previous experiments (1,3,5,6) have shown either that the growth rate is zero at a cavity size considerably above the critical nucleus size, or that the rate of increase in size decreases with increasing time. Both of these behaviors are illustrated in Figure 7. Thus, all previous measurements of cavity growth rate in ceramic systems have resulted in either a zero growth rate (3,5,6) or a growth rate that decreases with time (1,3,6); steady state growth has not been observed.

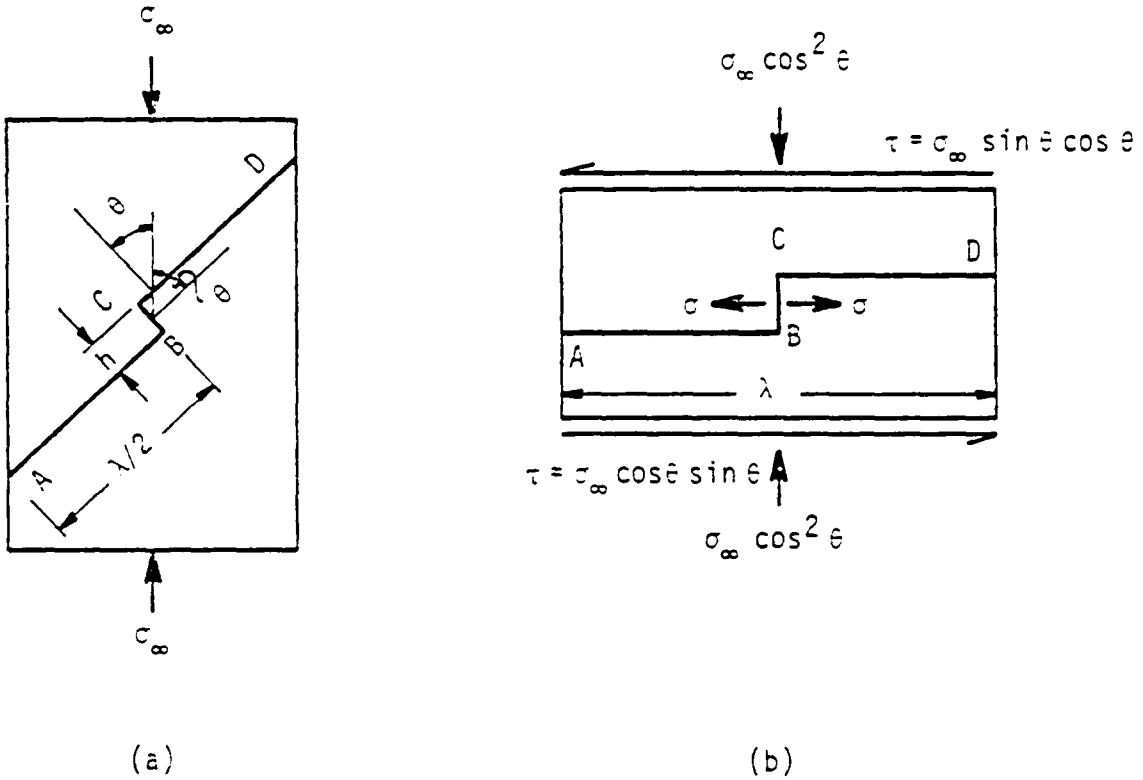


FIGURE 4. (a) SCHEMATIC OF AN INCLINED, FACETED GRAIN BOUNDARY (ABCD) WITH A LEDGE (BC), UNDER A COMPRESSIVE STRESS, σ_∞, AND (b) THE INDUCEMENT OF LOCAL TENSILE STRESSES AT GB LEDGE BC BY SLIDING OF BOUNDARY SEGMENTS AB AND CD.

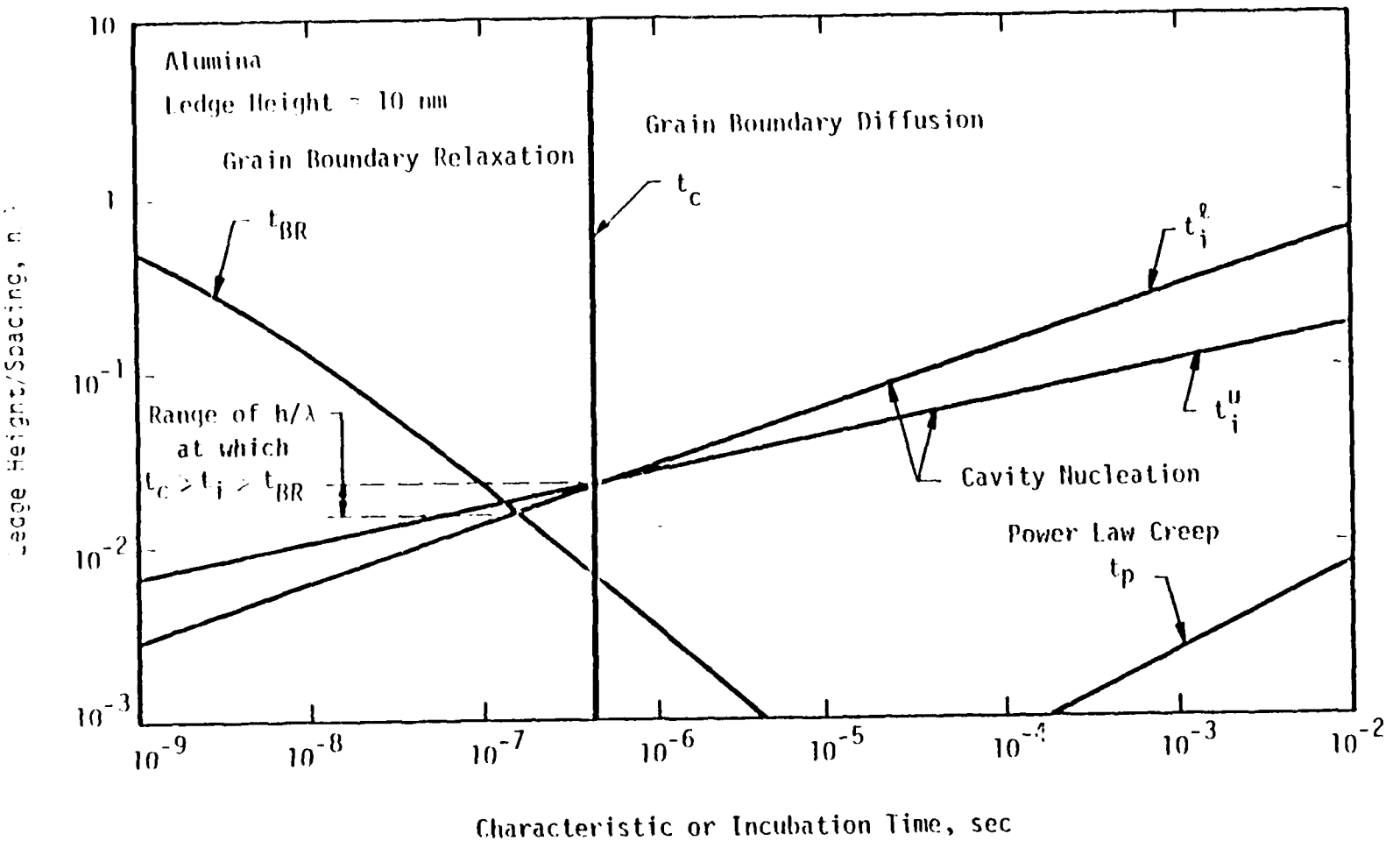


FIGURE 5. COMPARISON OF TWO ESTIMATES OF THE INCUBATION TIME FOR CAVITY NUCLEATION, t_i^{ℓ}, WITH THE CHARACTERISTIC TIMES FOR RELAXING STRESS CONCENTRATION AT A 10 NM HIGH GB LEDGE BY EITHER GRAIN BOUNDARY DIFFUSION, t_c, OR POWER-LAW CREEP, t_p. Also indicated is t_{BR}, the characteristic time for relaxing the shear stresses along sliding grain boundaries. The range of h/λ at which $t_c \geq t_i \geq t_{BR}$ occurs is $\approx 1\text{-}3\times10^{-2}$.

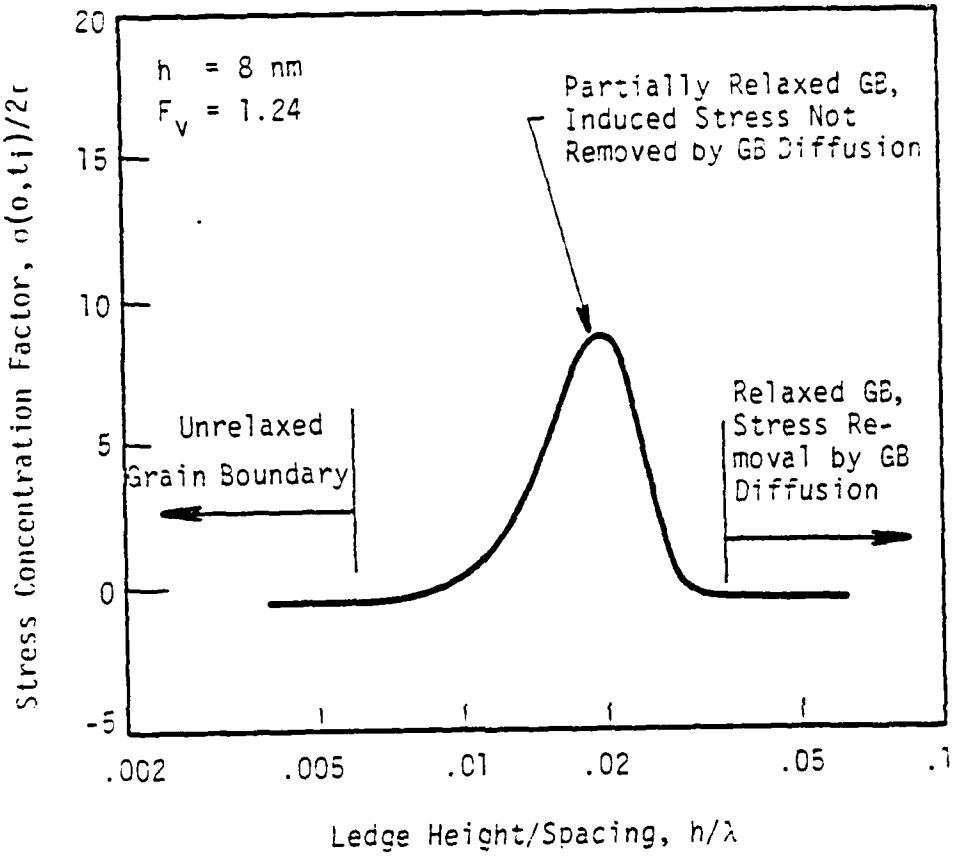


FIGURE 6. STRESS CONCENTRATION FACTOR AT AN 8 NM HIGH GB LEDGE AS A FUNCTION OF THE LEDGE HEIGHT TO SPACING RATIO, h/λ. The stress concentration factor is evaluated at $x = 0$ and $t = t_i$ for equilibrium-shaped cavities with $F_v = 1.24$; t_i is the incubation time for cavity nucleation.

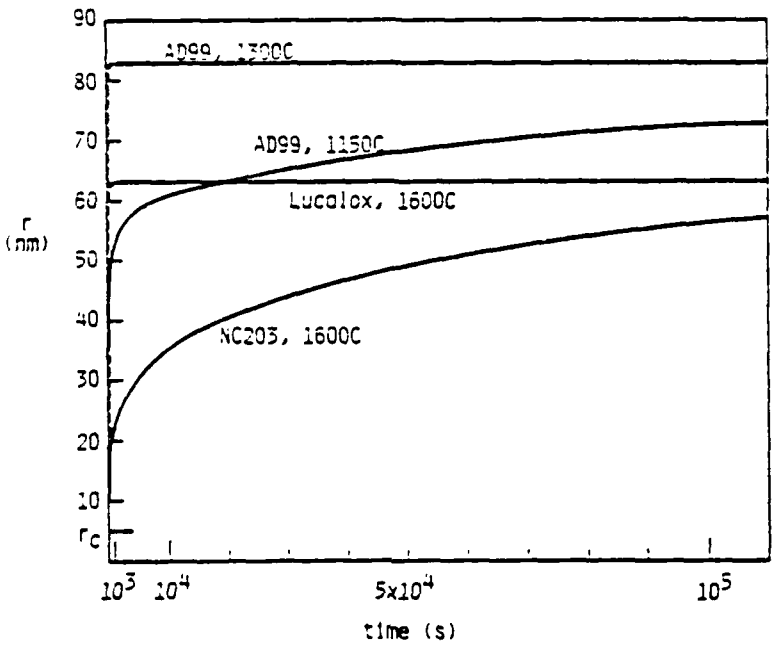


FIGURE 7. INCREASE IN INDIVIDUAL CAVITY RADIUS WITH TIME. The critical radius is indicated by r_c.

Cavity growth by grain boundary diffusion (pertinent to ceramics with "clean" grain boundaries) is described by (8)

$$\frac{dV}{dt} = \frac{2\pi\Omega Dh(\sigma_n - 2\gamma/R)}{kT} f(\ell/R) \tag{1}$$

where V is the cavity volume, t is time, Ω is the atomic volume, D is the grain boundary diffusion coefficient, h is the grain boundary height, σ_n is the normal stress across the boundary, γ is the surface energy, R is the cavity radius, ℓ is the cavity spacing, k is Boltzman's constant, and T is temperature. Similarly, the growth of a cavity by a viscous process (i.e., for ceramics with glassy grain boundary films) is described by (9)

$$\dot{R} = \frac{2\sqrt{3}(\ell^2 - \beta\pi R^2)\,\dot{h}}{2\pi R h\,\beta} \tag{2}$$

and

$$\dot{h} = \frac{h^3[\sigma_n - 2\gamma K(1-0.9\alpha^2]}{6\eta\,\ell^2[0.96\alpha^2 - \ln\alpha - 0.23\alpha^4 - 0.72]} \tag{3}$$

where α is the ratio of cavity radius to cavity spacing, η is the viscosity of the glassy phase, and K is a constant related to the ratio of the grain boundary, surface, and interfacial energies. In both descriptions of cavity growth the growth rate is proportional to the boundary normal stress. Thus, the experimental observation of transient cavity growth rates implies the existence of transient boundary tractions.

The subject of transient boundary tractions was discussed previously in dealing with cavity nucleation concepts. However, the characteristic

time for the relaxation of stress concentrations on boundary ledges is far too short to explain the long duration transients of Figure 7. Two other possibilities do exist, however. Raj (10) has identified and analyzed transient stresses resulting purely from the presence of the cavities and from grain boundary sliding transients, Figure 8. In both cases, the characteristic relaxation time, τ, is given by (10)

$$\tau = \frac{32(1-\nu^2)\ L^3 kT}{\pi^3 ED\ h\Omega} \tag{4}$$

where ν is Poisson's ratio, E is Young's modulus, and L is the characteristic diffusion length, which for the first case would be one-half of the cavity spacing and for the second case would be one-half of the grain size. The characteristic relaxation time for stress concentrations with a repeat distance of the cavity spacing is much too short to explain the growth transients which persist for a number of hours in both silicon carbide and alumina; however, the characteristic time for stress concentrations with a repeat distance equivalent to the grain size is of the same order as the duration of the growth transient. Hence, it appears that cavity growth may also be controlled by grain boundary sliding.

The above analysis of recent experimental studies of compression crept ceramics, supported by microstructural modeling, has identified a number of stochastic aspects of cavitation. The stochastic nature of cavitation arises primarily due to the dependence of both cavity nucleation and cavity growth on grain boundary sliding. A degree of randomness is also imposed by the nonuniform distribution of nucleation sites dictated by the narrow range of h/λ for which nucleation is likely. If

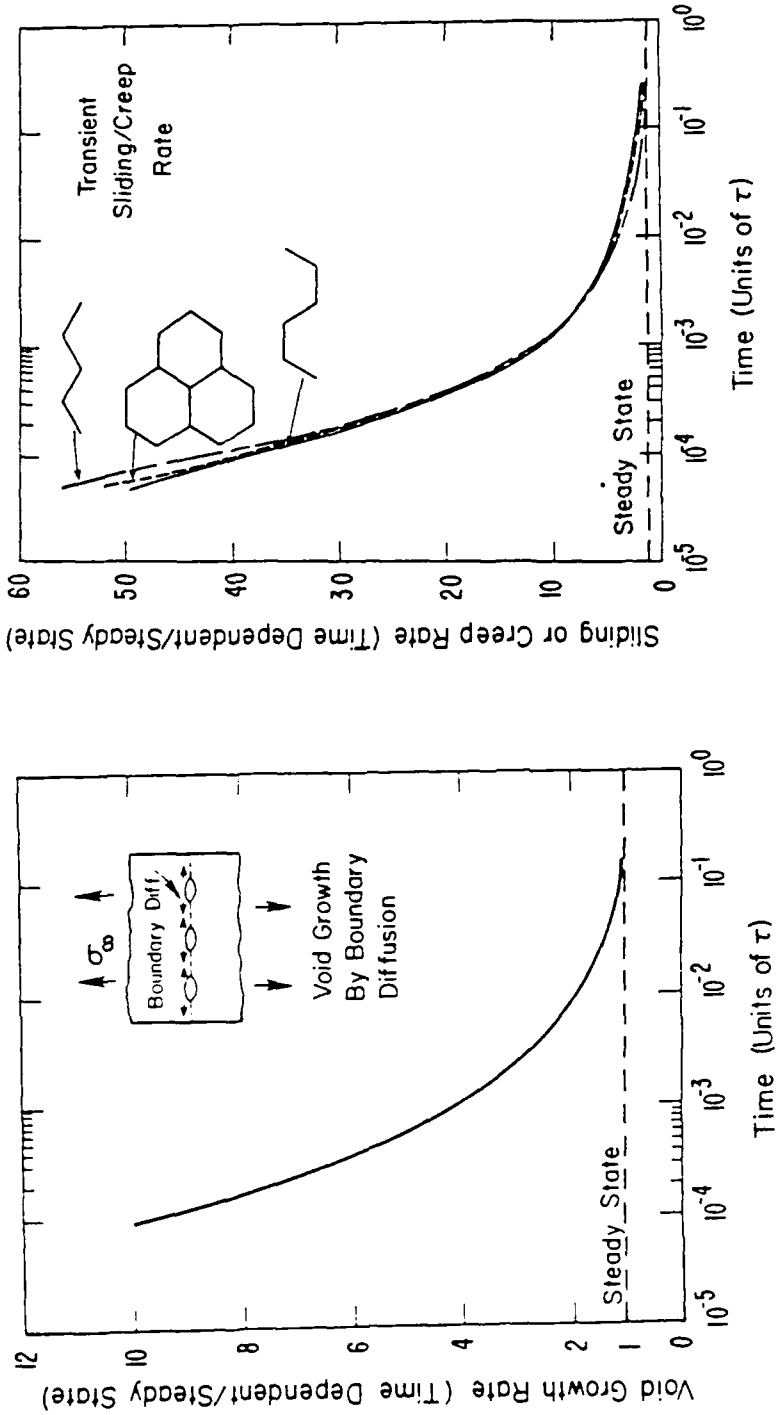


FIGURE 8. ILLUSTRATION OF THE VOID GROWTH AND GRAIN BOUNDARY SLIDING TRANSIENTS AS ANALYZED BY RAJ (10).

cavitation under pure tensile loading is governed by factors similar to those that control cavitation during compressive loading, the measurement of grain boundary sliding rates and their correlation with cavitation rates together with the development of a statistical model of cavitation will be crucial to the understanding and modeling of tensile creep failure. These tasks are currently in progress.

D. References

1. R. A. Page, J. Lankford, and S. Spooner, Acta Met. 32 1275 (1984).
2. R. A. Page and S. Spooner, J. Mat. Sci. 21 1417 (1986).
3. J. Lankford, K. S. Chan, and R. A. Page, Fracture Mechanics of Ceramics, R. C. Bradt, A. G. Evans, D. P. H. Hasselman, and F. F. Lange, eds., Plenum Press, New York, (in press).
4. R. A. Page and J. Lankford, J. Am. Ceram. Soc. 66 C-146 (1983).
5. R. A. Page, J. Lankford, and S. Spooner, J. Mat. Sci. 19 3360 (1984).
6. R. A. Page, J. Lankford, K. S. Chan, K. Hardman-Rhyne, and S. Spooner, J. Am. Ceram. Soc., (submitted).
7. K. S. Chan, R. A. Page, and J. Lankford, Acta Met., (in press).
8. M. V. Speight and W. Beere, Metal Sci. 9 190 (1975).
9. R. Raj and C. H. Dang, Phil. Mag. 32 909 (1975).
10. R. Raj, Met. Trans. 6A 1499 (1975).

III. PUBLICATIONS (AFOSR SPONSORSHIP)

1. "Stochastic Aspects of Creep Cavitation in Ceramics", R. A. Page and K. S. Chan, manuscript in preparation, April, 1986.

IV. PROGRAM PERSONNEL

Name	Title	
Dr. Richard A. Page	Senior Scientist	Co-Principal Investigators
Dr. James Lankford	Institute Scientist	
Dr. Kwai S. Chan	Senior Research Engineer	
Mr. Andrew Nagy	Senior Research Engineer	
Mr. Jerry L. Sievert	Staff Technician	
Mr. Forrest S. Campbell	Staff Technician	
Mr. Arthur E. Nicholls	Senior Technician	

V. INTERACTIONS

1. A paper entitled, "Stochastic Aspects of Creep Cavitation", by R. A. Page and J. Lankford, was presented at AIME Annual Meeting in New Orleans, LA. 3-6 March 1986.
2. Initiated discussions with Professors A. Saxena and S. Stock of Georgia Tech concerning possibility of using synchrotron source for mapping the location of large full-facet cavities. 5 March 1986.
3. Professor G. Pharr of Rice University visited SwRI to discuss cavitation and creep failure in materials containing a continuous viscous grain boundary phase. 11 March 1986.

END

DTIC

8-86